

Polyimide Composites Properties of RTM370 Fabricated by VARTM

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High Temperature Polyimide Composites Materials & Processing

- ◆ Conventional PMR-15, PMR-II-50, AFR-PE4 polyimide composites all require solvent-based prepregs for part fabrication ⇒ *time consuming, costly and the use of solvents and diamines are hazardous*
- ◆ Fabricate net-shape polymer matrix composites from resins in the melt via RTM or VARTM using preforms ⇒ *eliminate costly hand lay-up and hazard*
⇒ *produce 30% cost saving & 20% weight saving for complex parts*
⇒ *adaptable to automatic process*

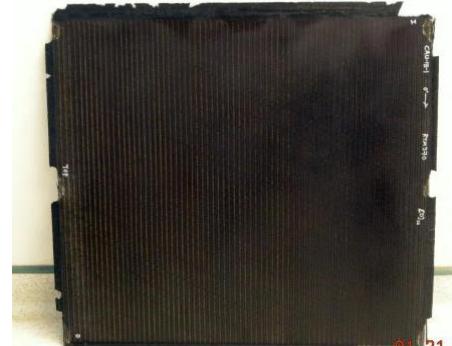
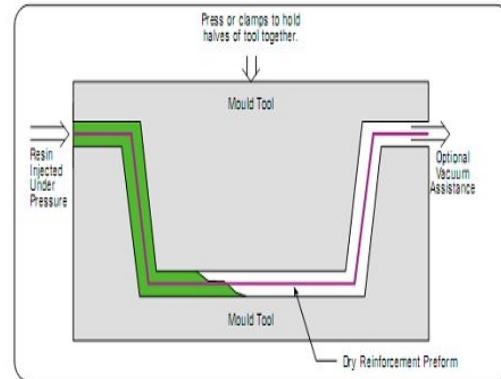
Challenge: RTM or VARTM requires low-melt viscosity that conventional polyimides derived from commercially available *s-dianhydride* ($> 10^3\text{-}10^5$ poise) cannot meet

- ◆ a-BPDA based imide resins have shown to exhibit low-melt viscosity (10-30 poise) at 280 °C ⇒ *amenable to low-cost RTM or VARTM process*
- ◆ Advance PMC temperature capability to 260-315°C beyond the state-of the-art RTM resins, such as epoxy (177 °C) & BMI (232 °C)

Objectives

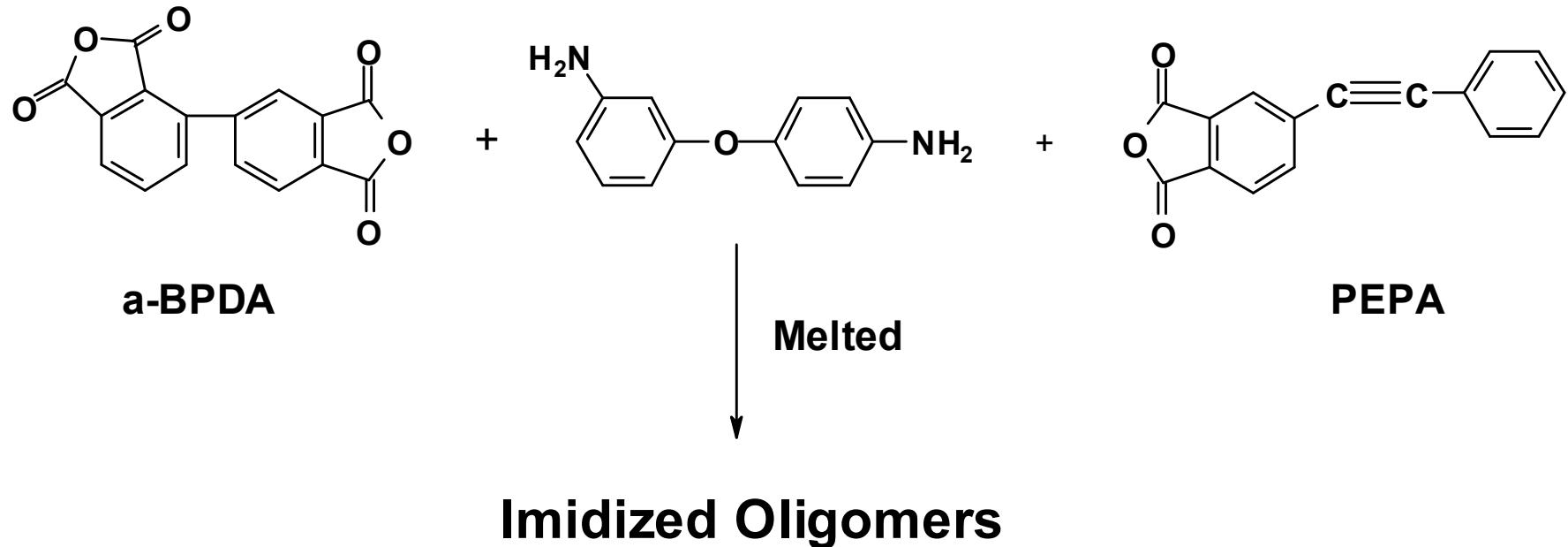
- ◆ Fabricated composite panels with RTM370 imide resin (~10-30 poise) by vacuum assisted resin transfer molding (VARTM)
- ◆ Compare mechanical properties of VARTM panels to RTM panels at 288-315°C (550-600°F)

VARTM vs RTM



VARTM	RTM
Use vacuum bag	Need a mold (expensive)
Use vacuum only	Use pressure and/or vacuum
15-20 psi	200 psi
Suitable for large part	Suitable for small part/mold

RTM370 Imide Resins



Advantages of imide resins containing a-dianhydrides:

- *Lower melt viscosities*
- *Higher T_g's*

Solvent-Free process:

- *No organic volatiles (Green)*
- *Adaptable to reactive extrusion (cost saving)*

Physical Properties of Imide Oligomers/ Resins Based on a-BPDA and 4-PEPA

Resin	Diamine	Oligomer Min. η @280 °C by Brookfield ¹ (Poise)	Oligomer Min. Complex $[\eta]^*$ @280°C ² (Poise)	Cured Resin T_g (°C) NPC ³ byTMA ⁵	Cured Resin T_g (°C) PC ⁴ @ 650°F
RTM370	3,4' -ODA	8.8	6.5	342	370 ⁵
RTM370 Composite		---	---	338 (DMA) ⁶	350 (DMA) ⁶

3,4' -ODA = 3,4' -Oxydianiline

¹ Absolute viscosity measured by Brookfield Viscometer at 280 °C.

² Complex viscosity measured by Aries Rheometer, using parallel plates.

³ NPC = No Post cure

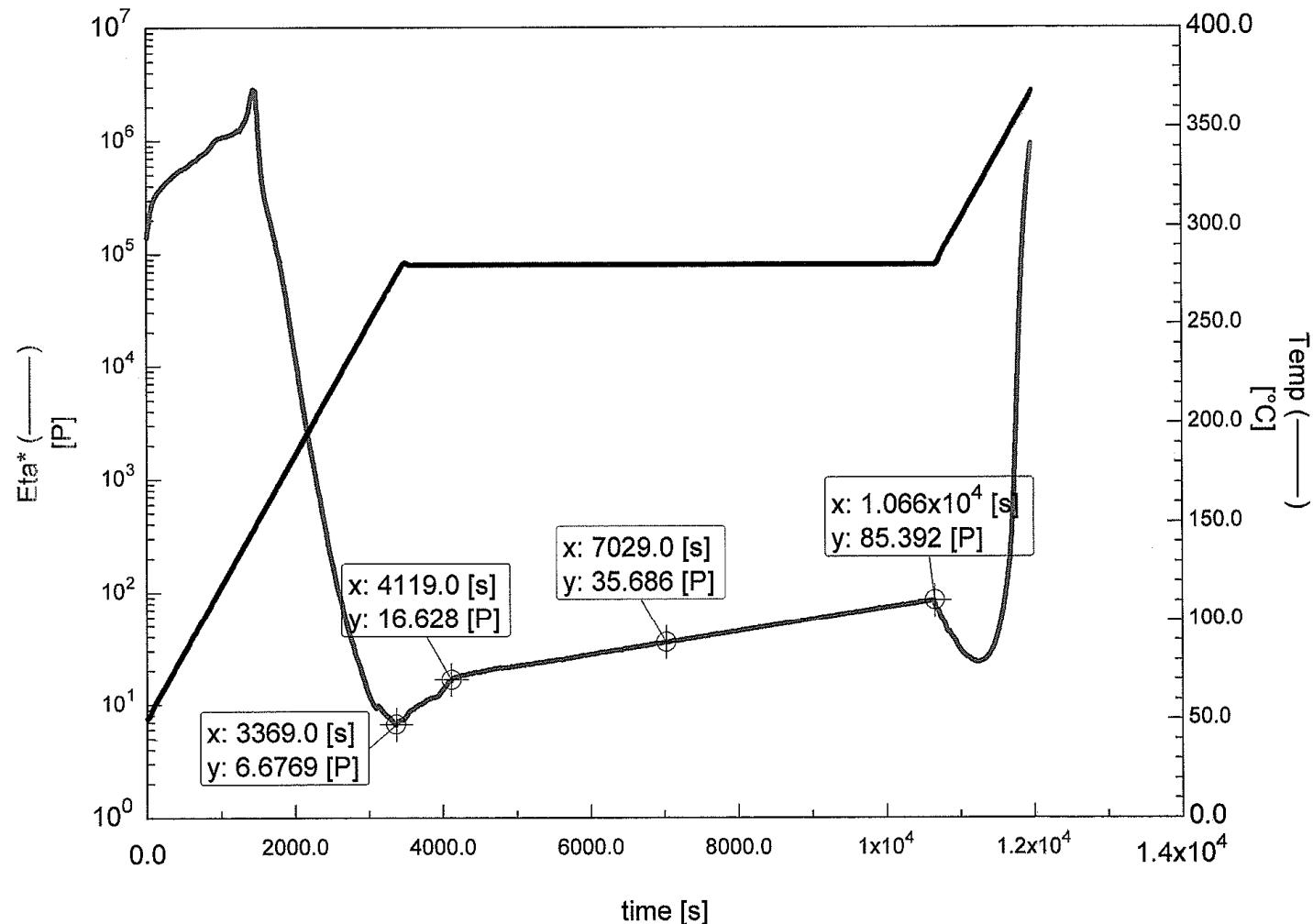
⁴ PC = Resin Postcured at 343 °C (650 °F) for 16 hrs while composite postcured@650°

⁵ TMA =Thermal mechanical analysis heated at 10 °C/min, using expansion mode.

⁶ DMA = Dynamic mechanical analysis were performed at 5 °C/min heating rate,
using single cantilever.

Rheology of APS's RTM370 Imide Resin

2 hr hold at 280 °C

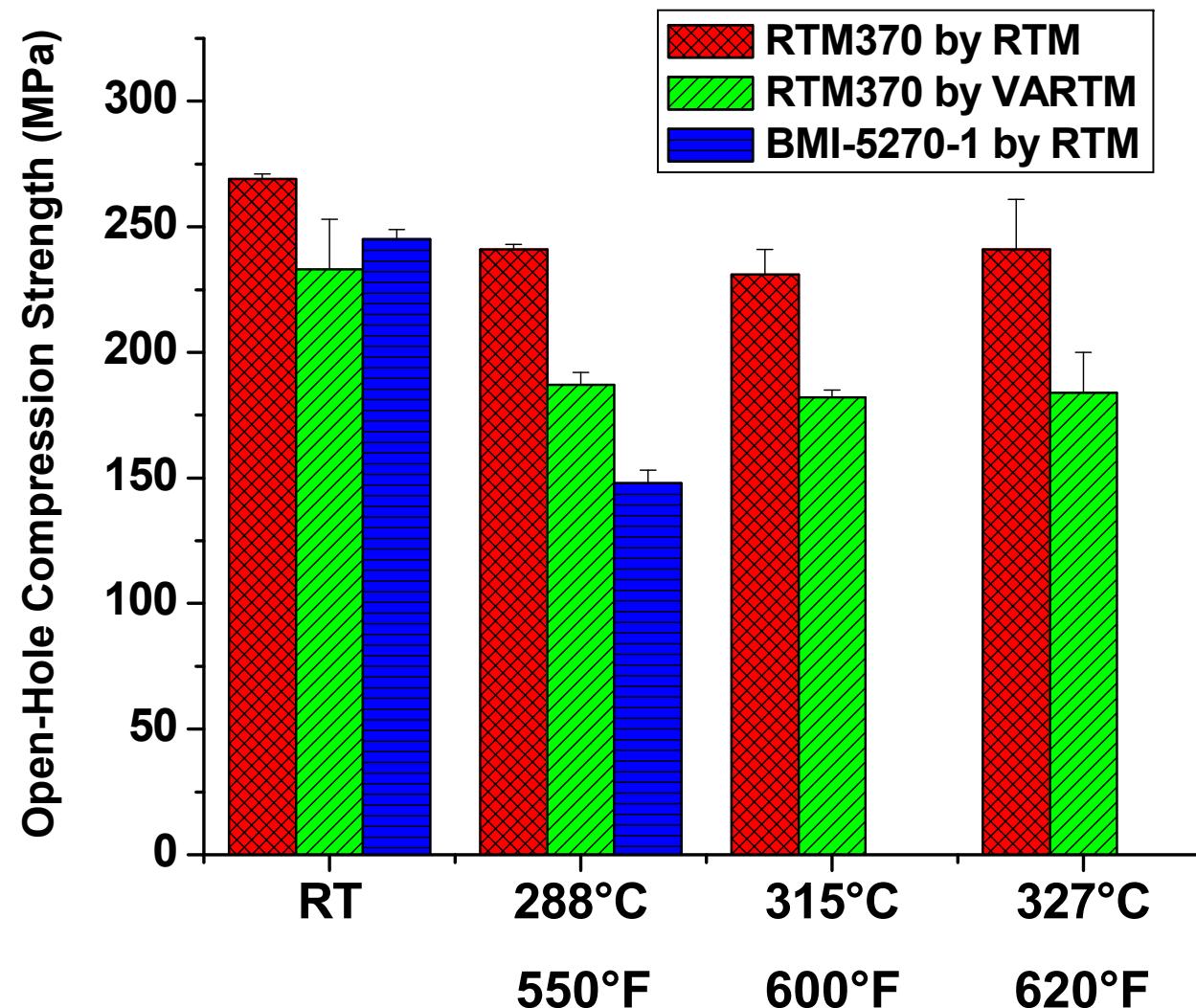


RTM370 Composite Property Comparison

VARTM vs RTM (T650-35/HT sizing)

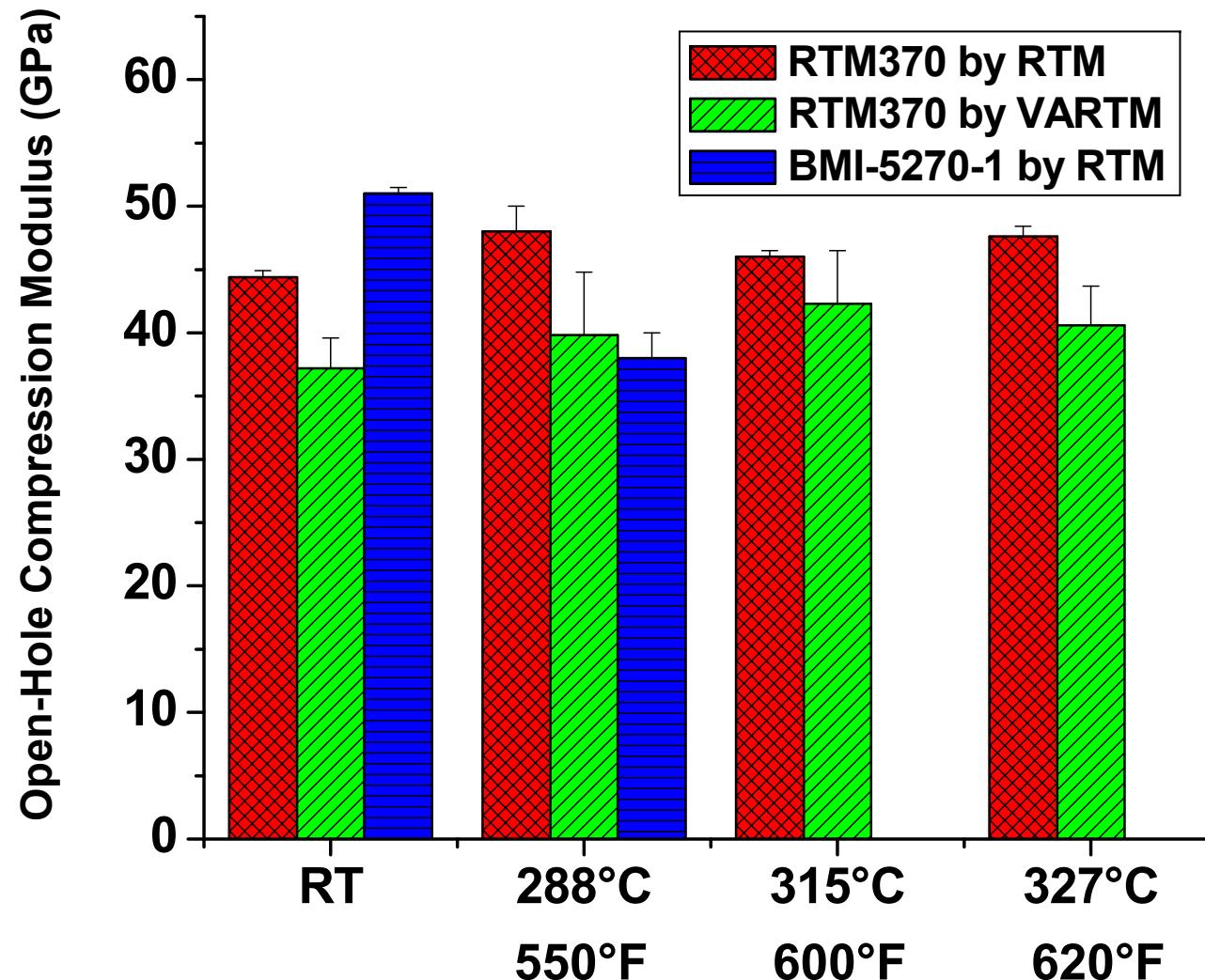
	Test Temp.	OHC Strength (MPa)			OHC Modulus (GPa)			SBS Strength (MPa)		
		Initial	500h @550°F 288°C	1000h @550°F 288°C	Initial	500h @550°F 288°C	1000h @550°F 288°C	Initial	500h @550°F 288°C	1000h @550°F 288°C
VARTM	RT	233	194	120	37	38	33	43	37	22
	288°C	186	197	135	40	42	38	31	27	17
	315°C	182	---	118	42	---	38	29	26	15
	327°C	184	---	---	41	---	---	30	---	16
RTM	RT	269	287	230	44	47	44	51	54	43
	288°C	242	244	198	48	44	45	41	41	41
	315°C	231	---	---	46	---	---	31	---	---
	327°C	241	---	---	48	---	---	30	---	---

RTM370 OHC Strength VARTM vs RTM (T650-35/8HS/HT Sizing)

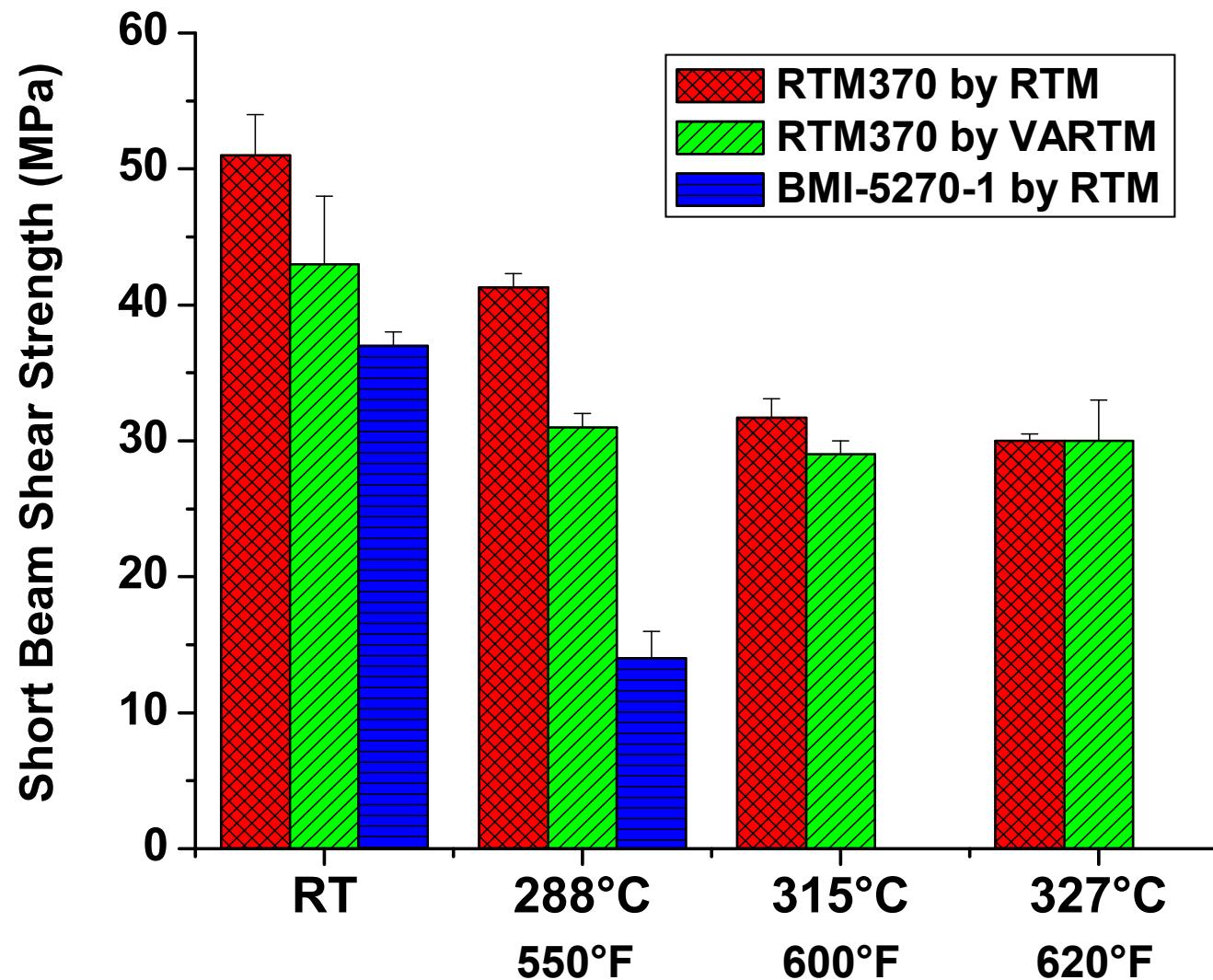


RTM370 OHC Modulus VARTM vs RTM

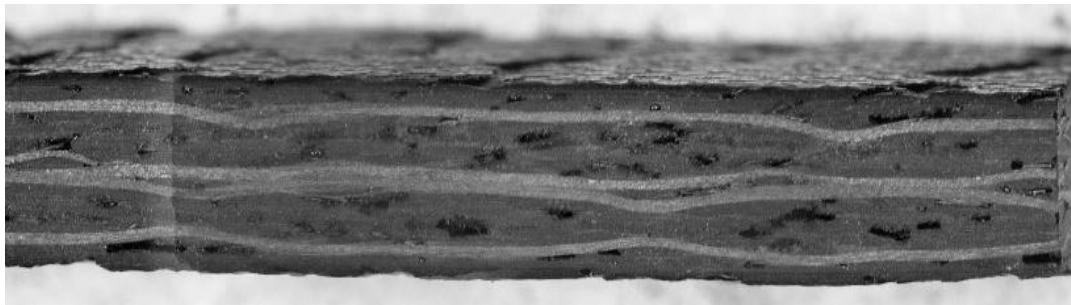
(T650-35/8HS/HT Sizing)



RTM370 SBS Strength VARTM vs RTM (T650-35/8HS/HT Sizing)



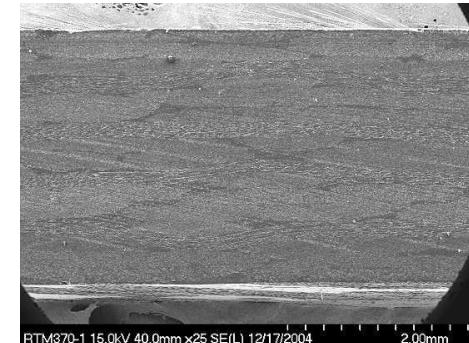
VARTM vs RTM



SEM of RTM370 made by VARTM

Void content = ~6.5% after postcured at 650°F/8 h

Resin vs fiber content = 47-50% vs 53-50%



RTM370 Made by RTM

Void content= ~1%

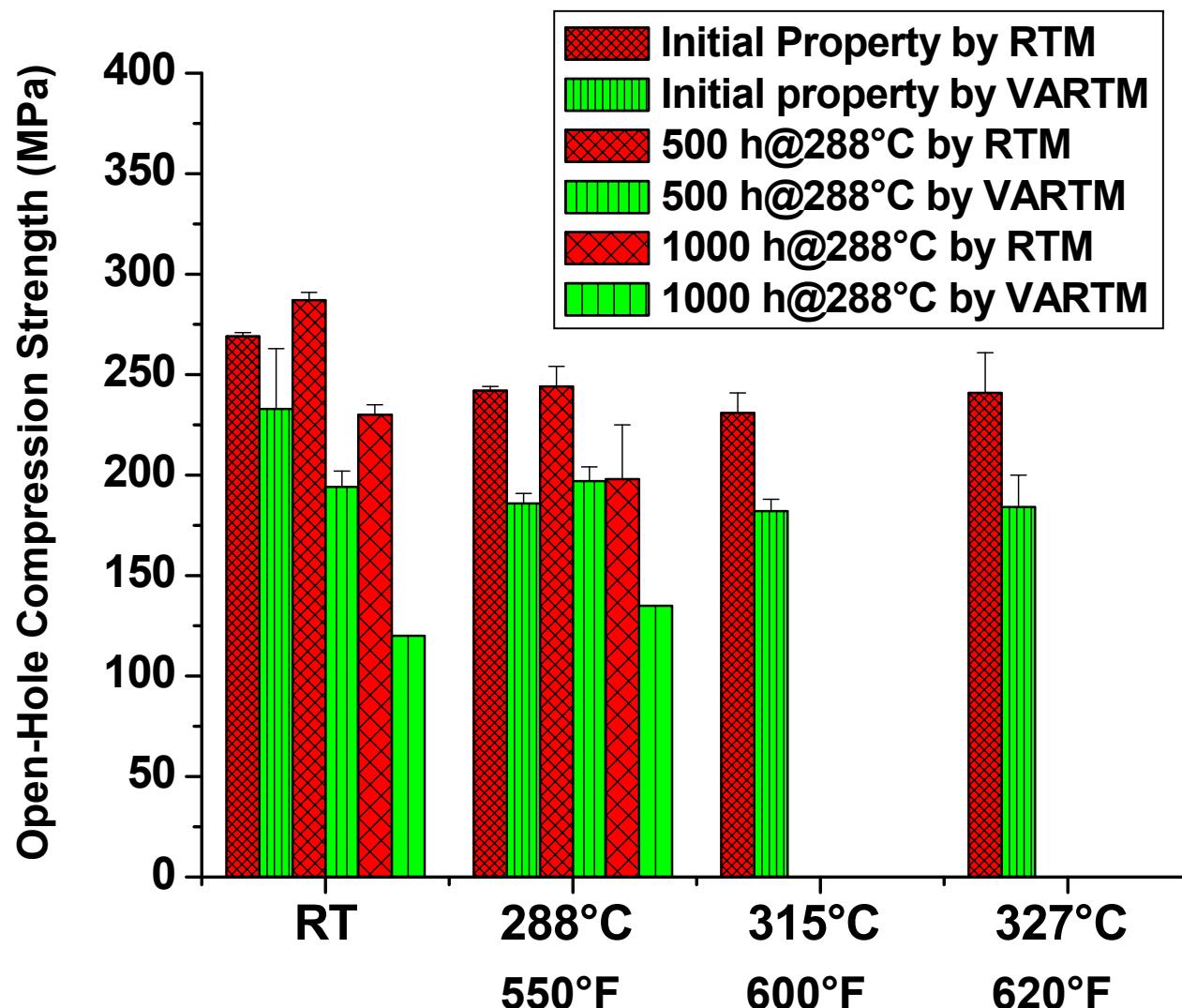
Resin content = 44-47%

VARTM	RTM
15-20 psi	200 psi
Higher void content	Lower void content

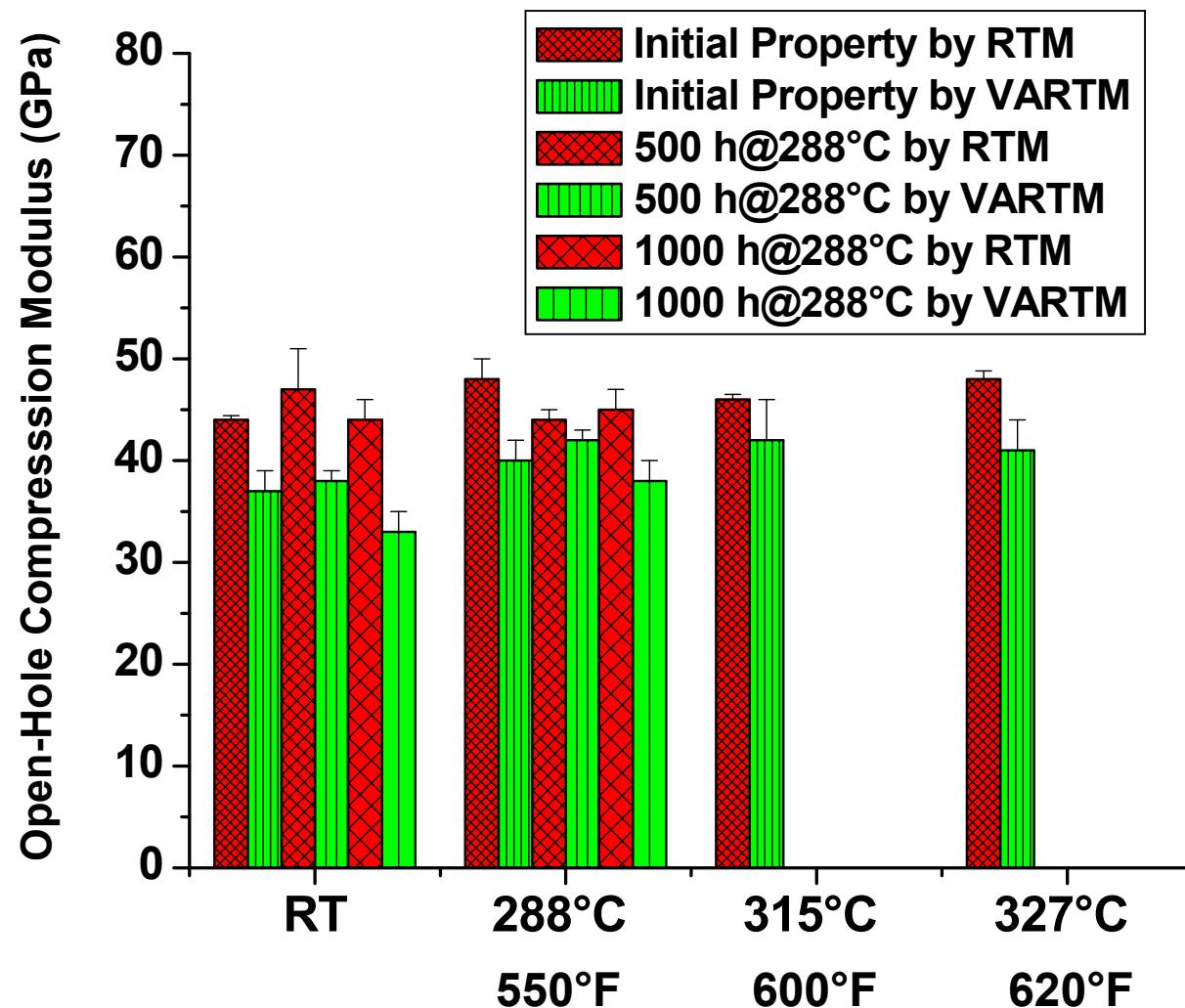
Process Improvement:

- ♦ Injection at 260°C instead of 280°C ⇒ Longer pot-life, but longer injection time
- ♦ Add hold time at ~300°C, instead of direct ramp from 280°C to 371°C

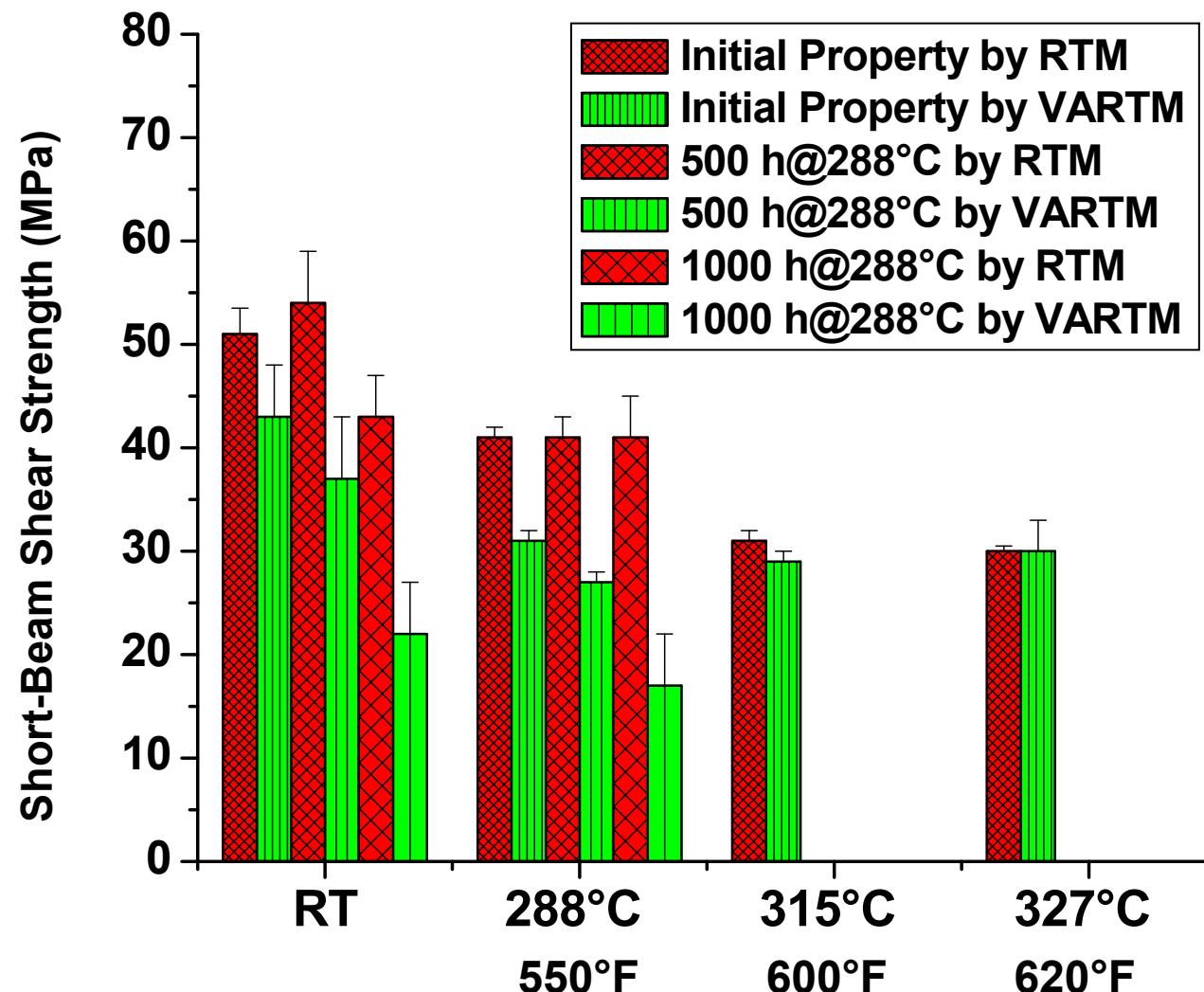
Open-Hole Compression Strength of RTM370 Composites Subjected to Isothermal Aging at 288°C for 1000 h in Air



Open-Hole Compression Modulus of RTM370 Composites Subjected to Isothermal Aging at 288°C for 1000 h in Air



Short-Beam Shear Strength of RTM370 Composites Subjected to Isothermal Aging at 288°C for 1000 h in Air



Summary

- ♦ Demonstrated RTM370 imide resin can be processed by VARTM out of autoclave
- ♦ VARTM panels have higher void content than those made by RTM, due to lack of pressure
 ⇒ VARTM panels appeared to have slightly lower OHC, but comparable SBS at high temp. & retained good mechanical properties after aging
- ♦ Need process development to reduce void content to < 2% for aerospace applications
- ♦ Need to improve the resin content of VARTM panels from 47-50% to 35-40%.

Acknowledgements